PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 : C08K 5/13 — A1

(11) International Publication Number: **WO 94/13736**

(43) International Publication Date: 23 June 1994 (23.06.94)

(21) International Application Number: PCT/EP93/03630

(22) International Filing Date: 14 December 1993 (14.12.93)

(30) Priority Data:
92311491.2 16 December 1992 (16.12.92) EP
(34) Countries for which the regional or international application was filed: GB et al.

(71) Applicant *(for CA only)*: SHELL CANADA LIMITED [CA/CA]; 400 - 4th Avenue S.W., Calgary, Alberta T2P 2H5 (CA).

(71) Applicant *(for all designated States except CA)*: SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V. [NL/NL]; Carel van Bylandtlaan 30, NL-2596 HR The Hague (NL).

(72) Inventors: ROYALL, Sven, Edward; 2 York Road, Waterloo, London SE1 7NA (GB). VAN DER VEER, Wijtze; Badhuisweg 3, NL-1031 CM Amsterdam (NL). VAN DER VENNET, Renee, Theresa; One Shell Plaza, 900 Louisiana, Houston, TX 77002 (US). COX, Michael, Joseph; One Shell Plaza, 900 Louisiana, Houston, TX 77002 (US). STONE, April, Carolyn; One Shell Plaza, 900 Louisiana, Houston, TX 77002 (US).

(81) Designated States: AT, AU, BB, BG, BR, BY, CA, CH, CZ, DE, DK, ES, FI, GB, HU, JP, KP, KR, KZ, LK, LU, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, UA, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

Published
With international search report.
Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: REFINED PETROLEUM WAX COMPOSITION

(57) Abstract

A refined petroleum wax composition comprises a refined petroleum wax and, as a stabilising agent, tocopherol and/or a derivative thereof. The refined petroleum wax may be a paraffin wax, a microcrystalline wax or a Fischer-Tropsch wax. A method for preparing the refined petroleum wax composition comprises adding to a refined petroleum wax tocopherol and/or a derivative thereof.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	**GB**	United Kingdom	**MR**	Mauritania
AU	Australia	**GE**	Georgia	**MW**	Malawi
BB	Barbados	**GN**	Guinea	**NE**	Niger
BE	Belgium	**GR**	Greece	**NL**	Netherlands
BF	Burkina Faso	**HU**	Hungary	**NO**	Norway
BG	Bulgaria	**IE**	Ireland	**NZ**	New Zealand
BJ	Benin	**IT**	Italy	**PL**	Poland
BR	Brazil	**JP**	Japan	**PT**	Portugal
BY	Belarus	**KE**	Kenya	**RO**	Romania
CA	Canada	**KG**	Kyrgyzstan	**RU**	Russian Federation
CF	Central African Republic	**KP**	Democratic People's Republic of Korea	**SD**	Sudan
CG	Congo			**SE**	Sweden
CH	Switzerland	**KR**	Republic of Korea	**SI**	Slovenia
CI	Côte d'Ivoire	**KZ**	Kazakhstan	**SK**	Slovakia
CM	Cameroon	**LI**	Liechtenstein	**SN**	Senegal
CN	China	**LK**	Sri Lanka	**TD**	Chad
CS	Czechoslovakia	**LU**	Luxembourg	**TG**	Togo
CZ	Czech Republic	**LV**	Latvia	**TJ**	Tajikistan
DE	Germany	**MC**	Monaco	**TT**	Trinidad and Tobago
DK	Denmark	**MD**	Republic of Moldova	**UA**	Ukraine
ES	Spain	**MG**	Madagascar	**US**	United States of America
FI	Finland	**ML**	Mali	**UZ**	Uzbekistan
FR	France	**MN**	Mongolia	**VN**	Viet Nam
GA	Gabon				

REFINED PETROLEUM WAX COMPOSITION

The present invention relates to a petroleum wax composition, in particular to a petroleum wax composition comprising a stabilising agent.

Refined petroleum waxes are valuable products finding use in such applications as candles, paper coatings, foodstuffs and protective sealants for foodstuffs, adhesive compositions, polishes and electrical insulation. The waxes are mixtures of hydrocarbons having high molecular weights and are solid at ambient conditions of temperature and pressure.

The refined petroleum waxes are most preferably obtained as white, translucent, odourless materials. However, it has been found that, upon exposure to oxygen, for example by contact with air, petroleum waxes degrade, resulting in the waxes becoming coloured and odorous. Accordingly, it is common practice to add to the finished waxes a stabilising agent.

Thus, US patent number 4,766,166 (US-A-4,766,166) discloses a wax composition comprising a synthetic wax, such as a Fischer-Tropsch wax, in combination with a high molecular weight polyethylene material. It is disclosed in US-A-4,766,166 that, in addition to the synthetic wax and the polyethylene components, the composition may contain other ingredients including antioxidants. Butylated hydroxy toluene (BHT) is given as a specific example of an antioxidant.

US patent number 4,471,086 (US-A-4,471,086) is directed to an adhesive composition comprising a Fischer-Tropsch hydrocarbon wax, which composition may contain one or more antioxidants. Effective antioxidants disclosed in US-A-4,471,086 are tris(di-t-butyl-p-hydroxybenzyl)-trimethyl benzene, alkylated bisphenol, zinc dibutyl dithiocarbamate, 4,4-methylene bis(2,6-di-tert-butyl-phenol), tetrakis[methylene(3,5-di-tert-butyl-4-hydroxyhydro-

cinamate)methane], lauryl stearyl thiodipropionate, dilauryl 3,3-thiodipropionate and 2,6-di-tert-butyl-p-cresol (BHT).

Recently, however, there has been a growing desire to reduce the use of compounds such as BHT as antioxidants in wax compositions, in particular when used in food or food-related applications. Accordingly, there is a need for compounds which are effective as antioxidants in petroleum wax compositions and which offer alternatives to compounds such as BHT. Tocopherol (commonly referred to as vitamin E) is a naturally occurring product present in many foodstuffs. Surprisingly, it has been found that tocopherol and derivatives thereof, when included in compositions of petroleum wax, are very effective as antioxidants.

Tocopherol is known to be effective as an antioxidant when used in certain products. Thus, the texts of European Patent applications published under the numbers EP-A-0 459 354 and EP-A-0 464 491 disclose lubricating base oil compositions comprising liquid paraffins and fatty acids. The compositions may comprise one or more dibutylhydroxytoluenes, for example BHT, and/or tocopherol as antioxidants. Lubricating base oil compositions comprising tocopherol as an antioxidant are disclosed in Japanese patent applications published under the numbers J5 4099-104 and J5 6053-188.

A raw oil for use in formulating hydraulic fluids is disclosed in International Patent application publication number WO 91/02784, which oil comprises a refined rapeseed and/or soya oil, selected esters, selected monocarboxylic acids and/or fatty acids, and an antioxidant selected from the group methoxyphenol, ethoxyphenol, butylhydroxyanisol, butylhydroxytoluene, methoxyhydroquinone, ethoxyhydroquinone, tert-butylhydroquinone and/or tocopherol.

Further, German patent numbers DE 1114319 and DE 1136102 disclose polyolefin compositions, in particular polyethylene and polypropylene compositions, comprising tocopherol and a naturally occurring carboxylic acid.

Finally, Shmulovich et al., Kinetika i Kataliz, Vol. 19, No. 2, pp. 501 to 503, March-April, 1978, describe tests conducted

in the performance of tocopherol, ionol, beta-naphthol and butyl-hydroxyanisole as antioxidants for liquid petrolatum (medicinal vaseline oil).

Surprisingly, it has been found that refined petroleum wax compositions comprising tocopherol and/or a derivative thereof exhibit a very high resistance to oxidation. This results in the wax composition retaining a high quality of colour and remaining substantially odourless for extensive periods of time. More surprisingly, it has been found that refined petroleum wax compositions comprising a refined petroleum wax and tocopherol as an antioxidant exhibit a substantially greater resistance to oxidation than similar compositions comprising equivalent amounts of conventionally applied antioxidants, for example BHT.

Accordingly, the present invention provides a refined petroleum wax composition comprising a refined petroleum wax and, as a stabilising agent, tocopherol and/or a derivative thereof.

Tocopherol and its derivatives are well known, naturally occurring compounds. For details of the structure of the compounds reference is made to the Dictionary of Organic Compounds, Fifth Edition, published by Chapman and Hall. As used herein, the term "tocopherol" is a reference to one or a mixture of the tocopherols, including α-tocopherol (5,7,8-trimethyltocol), β-tocopherol (5,8-dimethyltocol), γ-tocopherol (7,8-dimethyltocol), n-tocopherol (7-methyltocol) and Γ-tocopherol (5,7-dimethyltocol). The term "derivative" when used in association with the tocopherols is a reference to any derivative of tocopherol in which a stearically hindered hydroxyl group on the aromatic ring of the molecule is present. Examples of derivatives include the tocotrienols. Preferably the composition comprises a tocopherol, with α-tocopherol being particularly preferred. Tocopherol and derivatives thereof may exist in a variety of isomeric forms. Tocopherol and/or derivatives thereof may be present in the composition in either one or a mixture of isomeric forms.

The tocopherols and their derivatives are are available commercially, for example from the Sigma Chemical Company Ltd.,

United Kingdom.

Tocopherol and/or a derivative thereof is present in the composition in an amount sufficient to be effective in preventing oxidation of the wax. Typically, the composition comprises tocopherol and/or a derivative thereof in an amount of up to 5% wt, more preferably up to 1% wt. Most surprisingly, it has been found that tocopherol and/or its derivatives may be present in the composition in only very small amounts and yet still be effective as an antioxidant. Thus, the composition may comprise tocopherol and/or a derivative thereof in an amount of up to 200 ppm, more preferably up to 100 ppm, especially up to 50 ppm. It has been found that tocopherol and/or a derivative thereof present in an amount of up to 15 ppm is very effective as an antioxidant for petroleum wax compositions.

The present invention relates to refined petroleum wax compositions. For the purposes of this specification, the term "petroleum wax" is a reference to both paraffin waxes and microcrystalline waxes as well as to Fischer-Tropsch waxes and other synthetic waxes having similar properties. Synthetic waxes, such as the high pressure and low pressure polyethylene waxes, characterised by, for example, very high melt viscosities, are not suitable for inclusion in the compositions of the present invention. Typical melt viscosities for the refined petroleum waxes are in the range of up to 15 cSt.

The refined petroleum waxes for inclusion in the composition are to be distinguished from the unrefined waxes, such as slack waxes, scale waxes and petrolatum, and the liquid petroleum waxes. The unrefined waxes and liquid petroleum waxes may be characterised by their high oil content, the oil content of these materials being above 5% wt, and typically much higher, for example between 10 and 20% wt. The oil content of the wax may be determined using ASTM method D 721.

For a general discussion of refined petroleum waxes, reference is made to Ullmanns Encyklopädie der technischen Chemie, 4th Edition, Volume 24.

Paraffin waxes are petroleum waxes consisting principally of alkanes. They are macrocrystalline, brittle solids, typically comprising from about to 40 to 90% wt normal alkanes, the remainder being isoalkanes and cycloalkanes. Paraffin waxes have a low affinity for oil, the oil content being typically less than 2% wt, more typically less then 1% wt. Paraffin waxes are obtained from the conventional refining of crude oil and are typically recovered from a distillate by solvent extraction. Paraffin waxes suitable for inclusion in the compositions of the present invention include the crude hard waxes and raw waxes, as well as the semi-refined, filtered and fully-refined waxes. Preferred paraffin waxes for inclusion in the composition are the semi-refined, filtered and fully-refined waxes, especially the fully-refined waxes.

Microcrystalline waxes are petroleum waxes containing substantial quantities of hydrocarbons other than normal alkanes. The oil content of the microcrystalline waxes is higher than that of the paraffin waxes, typically 1 to 5% wt. Microcrystalline waxes are produced during the conventional refining of crude oil and are typically recovered from the residual fraction of crude oil distillation. Suitable microcrystalline waxes for inclusion in the composition include the plastic microwaxes and the hard microwaxes.

The refined petroleum wax composition may also comprise a synthetic wax. For the purposes of this specification, references to a synthetic wax are references to a Fischer-Tropsch wax or waxes having similar properties. Fischer-Tropsch waxes may be produced from a mixture of carbon monoxide and hydrogen (often referred to as synthesis gas) by the Fischer-Tropsch synthesis. Suitable processes for producing the Fischer-Tropsch waxes are well known in the art.

In the Fischer-Tropsch synthesis, a mixture of carbon monoxide and hydrogen is contacted at elevated temperature and pressure with a suitable catalyst. Catalysts for use in the Fischer-Tropsch synthesis process frequently comprise, as the catalytically active component, a metal from Group VIII of the Periodic Table of the Elements. Particular catalytically active metals include

ruthenium, iron, cobalt and nickel. Especially preferred waxes for inclusion in the composition of the present invention are those prepared by a Fischer-Tropsch synthesis process employing a catalyst comprising cobalt as the catalytically active component.

The catalytically active metal is preferably supported on a porous carrier. The porous carrier may be selected from any of the suitable refractory metal oxides or silicates or combinations thereof known in the art. Particular examples of preferred porous carriers include silica, alumina, titania and mixtures thereof.

If desired, the catalyst may also comprise one or more metals or metal oxides as promoters. Suitable metal oxide promoters may be selected from Groups IIA, IIIB, IVB, VB and VIB of the Periodic Table of the Elements. Preferred metal oxide promoters are oxides of the metals of Group IVB, in particular titanium and zirconium. Suitable metal promoters may be selected from Groups VIIB and VIII of the Periodic Table. Platinum and palladium are especially preferred metal promoters.

Examples of suitable catalysts for preparing waxes by the Fischer-Tropsch synthesis are disclosed in European patent applications publication numbers EP-A-0 104 672, EP-A-0 110 449, EP-A-0 127 220, EP-A-0 167 215, EP-A-0 180 269 and EP-A-0 221 598.

The Fischer-Tropsch synthesis is typically conducted at a temperature of from about 125 to about 350 °C, preferably from about 175 to 250 °C. Typical operating pressures for the synthesis are in the range of from about 5 bar to about 100 bar, more preferably from about 10 to about 50 bar. During the process, the catalyst is typically contacted with a gaseous mixture comprising hydrogen and carbon monoxide in a ratio of less than 2.5, preferably less than 1.75. More preferably, the hydrogen to carbon monoxide ratio of the mixture is in the range of from 0.4 to 1.5, especially from 0.9 to 1.3.

Waxes produced in the Fischer-Tropsch synthesis are present substantially in the liquid phase under the conditions prevailing in the process. However, the effluent from the process comprises, in addition to the desired waxes, unconverted feed gas and lighter

gaseous and liquid components produced during the synthesis reactions. The waxes may be separated from the unconverted feed gas and lighter components by conventional separation techniques well known in the art, for example distillation.

The waxes, once separated from the lighter components may then, if desired, be further refined to yield one or more wax fractions. Refining techniques for the waxes are known in the art. Owing to the low thermal stability of the waxes, high temperatures should be avoided. Accordingly, separation techniques such as vacuum distillation should be applied using, for example, the fine vacuum or short-path evaporators known in the art. Typical evaporating equipment for use in refining the waxes include the high vacuum falling film evaporators and the high vacuum wiped film evaporators. Typical operating pressures for the aforementioned evaporators range from 10^{-1} to 10^{2} Pa.

Fischer-Tropsch waxes consist almost entirely of paraffins and typically comprise at least 70% wt normal paraffins, more preferably greater than 80% wt normal paraffins. Fischer-Tropsch waxes comprising more than 90% wt normal paraffins are very suitable for use in the compositions of the present invention. The oil content of the Fischer-Tropsch waxes is typically less than 5% wt, more typically less than 3% wt. Fischer-Tropsch waxes having an oil content of less than 1% wt are especially suitable for inclusion in the compositions of the present invention.

At the very least, the compositions of the present invention consist of a refined petroleum wax and tocopherol and/or a derivative thereof. The composition may, however, comprise one or more other additives or stabilisers, if required. The composition comprises the refined petroleum wax (on an oil-free basis) in an amount of at least 90% wt, more preferably at least 95% wt, the remaining portion of the composition comprising any oil present in the wax, tocopherol and any further additives or stabilisers which may be present.

In a second aspect, the present invention is directed to the use of tocopherol and/or a derivative thereof as a stabiliser for a

refined petroleum wax.

The tocopherol and/or derivative thereof to be included in the petroleum wax composition may be added to the refined petroleum wax after final processing. Accordingly, in a further aspect, the present invention is directed to a process for preparing a petroleum wax composition, comprising adding to a refined petroleum wax tocopherol and/or a derivative thereof. The tocopherol and/or derivative thereof is conveniently added to the refined petroleum wax with the wax in the molten state. The resulting refined petroleum wax composition may be allowed to solidify or, alternatively, for ease of handling and transport may be maintained as a liquid.

The compositions of the present invention may be used in any of the applications of conventional refined wax compositions. Examples of which have been given in the introduction hereto. The compositions of the present invention are particularly suitable for use in food or food-related applications.

The present invention will be further explained by way of the following, illustrative examples.

Example 1

A refined Fischer-Tropsch wax was prepared using the following general procedure:

A cobalt/zirconium/silica catalyst was prepared following the procedure described in European Patent Application publication No. 0 428 223. The catalyst was loaded into a reaction vessel and reduced by contacting the catalyst with a mixture of hydrogen and nitrogen at a temperature of 250 °C, a pressure of 5 bar and a gas hourly space velocity of from 500 to 600 Nl/l/h. The activated catalyst was then contacted with a mixture of carbon monoxide and hydrogen having a hydrogen/carbon monoxide ratio of 1.1 at a gas inlet pressure of from 37 to 39 bar, a temperature of from 210 to 220 °C and a gas hourly space velocity of from 1110 to 1130 Nl/l/h to yield a hydrocarbon product.

The hydrocarbon product was subjected to a conventional distillation to remove the C_{17}- components, leaving a C_{18}+ hydro-

carbon mixture. The C_{18}+ hydrocarbon mixture thus produced was fed to a vertical wiped film evaporator of the SAMVAC type, operated to yield a C_{27}- light fraction and a C_{28}+ heavy fraction. The heavy fraction was fed to a second SAMVAC vertical wiped film evaporator, operated to yield a C_{40}- light fraction and a C_{41}+ heavy fraction. The C_{40}- fraction, having the properties set out in Table 1 below, was retained for further testing.

Table 1

Refined Fischer-Tropsch Wax

Carbon Distribution		
$<C_{22}$	(% wt)	0.17
$<C_{28}$	(% wt)	5.8
C_{28} to C_{40}	(% wt)	90.9
$<C_{40}$	(% wt)	3.3
Oil Content (% wt)		0.6
Congealing Point (°C)		68.3
Viscosity (mm^2/s at 100 °C)		5.24

Two samples of the refined Fischer-Tropsch wax were heated to form a liquid and were blended with α-tocopherol in a concentration of 15 ppm and 50 ppm respectively. The resulting compositions were stored in an atmosphere of air at a constant temperature of 80 °C. At regular periods, the odour of the two samples was determined according to the method set out in ASTM D 1833. For comparative purposes, the odour of a third sample of the refined wax stored under the identical conditions but without the presence of an antioxidant, was also determined. The results of the storage test are set out in Table 2 below.

Table 2

Refined Fischer-Tropsch Wax Storage Tests

	ODOUR (rating 0 - 4; ASTM D 1833)		
α-tocopherol (ppm)	15	50	zero
Storage Time (weeks)			
1	0.5	0.5	1.0
2	0.5	0.5	1.5
3	0.5	0.5	1.5
5	0.5	0.5	2.5
12	1.0	0.5	3.0

Using the same test procedure as outlined above, a further three compositions comprising the refined Fischer-Tropsch wax were tested for stability during storage under air at 80 °C. At regular intervals, the concentration of aldehydes/ketones, acids/anhydrides and esters accumulated in the wax was determined using Fourier Transform Infrared Spectroscopy at 120 °C in dry air, measuring absorption in the -OH band over a range of 3600 to 3500 cm^{-1} and the =O band over a range of from 1850 to 1650 cm^{-1}. At the same time, the peroxide number of the wax was determined using the Technical Association of the Pulp and Paper Industry (TAPPI)

peroxide number method T-659 (ASTM D 1832-87). The concentrations of these various oxygen-containing compounds is an indicator of the degree of oxidation of the composition. The results, in terms of the quantities of oxygen moieties detected, are given in Table 3 below.

Example 2

A crude-derived petroleum wax (commercially available after bauxite percolation and stabilisation as SHELLWAX 200 (trademark)) was prepared by solvent dewaxing and deoiling a high viscosity index lubricating base oil obtained by the conventional refining of crude oil. The wax had a viscosity at 100 °C of 4.2 cSt, an oil content of 0.1% wt, a melting point of 61 °C and a specific gravity at 15 °C of 0.917.

To a first sample of the wax was added α-tocopherol to a concentration of 15 ppm. To a second sample of the wax was added butylated hydroxytoluene (BHT, commercially available under the trademark IONOL) to a concentration of 15 ppm. For comparative purposes, a third sample was prepared without additives. The three compositions were maintained at a constant temperature of about 93 °C (200 °F) under an atmosphere of air. In order to measure the rate of oxidation of the wax, the peroxide number of the three samples was monitored using the Technical Association of the Pulp and Paper Industry (TAPPI) peroxide number method T-659 (ASTM D 1832-87). All the samples had an initial peroxide number of zero. The time taken for each sample to develop a significant peroxide number is given in Table 4 below.

Table 3

Refined Fischer-Tropsch Wax Storage Tests

		OXYGENATES			
	α-tocopherol (ppm)	Peroxides (meq/1000g)	Aldehydes/ Ketones (ppm)	Acids/ Anhydrides (ppm)	Esters (ppm)
Storage Time (weeks)					
0	0	3	<5	<5	<5
	15	<1	<5	<5	<5
	50	<1	<5	<5	<5
1	0	12	10	<5	<5
	15	<1	<5	<5	<5
	50	<1	<5	<5	<5
2	0	19	10	<5	<5
	15	<1	<5	<5	<5
	50	<1	<5	<5	<5
3	0	40	25	15	<5
	15	<1	<5	<5	<5
	50	<1	<5	<5	<5
5	0	68	75	40	<5
	15	<1	<5	<5	<5
	50	<1	<5	<5	<5
12	0	230	430	190	30
	15	<1	<5	<5	<5
	50	<1	<5	<5	<5

Table 4

Crude-derived Petroleum Wax Storage Tests

Sample No.	Additive	Additive (ppm)	Time taken to develop peroxide no. (days)
1	α-tocopherol	15	35
2	BHT	15	16
3	none	-	1

C L A I M S

1. A refined petroleum wax composition comprising a refined petroleum wax and, as a stabilising agent, tocopherol and/or a derivative thereof.

2. A composition according to claim 1, characterised in that the tocopherol is α-tocopherol, β-tocopherol, γ-tocopherol, n-tocopherol or Γ-tocopherol, preferably α-tocopherol.

3. A composition according to either of claims 1 or 2, characterised in that tocopherol and/or a derivative thereof is present in an amount of up to 5% wt, preferably up to 1% wt, more preferably up to 100 ppm, especially up to 50 ppm.

4. A composition according to any preceding claim, characterised in that the refined petroleum wax is a paraffin wax, preferably a semi-refined, filtered or a fully-refined waxes, especially a fully-refined wax.

5. A composition according to any one of claims 1 to 3, characterised in that the refined petroleum wax is a micro-crystalline wax, preferably a plastic microwax or a hard microwax.

6. A composition according to any one of claims 1 to 3, characterised in that the refined petroleum wax is a synthetic wax, preferably a Fischer-Tropsch wax.

7. A composition according to claim 6, characterised in that the refined petroleum wax is a Fischer-Tropsch wax having a normal-paraffin content of at least 70% wt, preferably at least 80% wt, more preferably at least 90% wt.

8. A composition according to any preceding claim, characterised in that the refined petroleum wax, on an oil-free basis, is present in an amount of at least 90% wt, more preferably 95% wt.

9. A method for preparing a composition as claimed in any preceding claim, comprising adding to a refined petroleum wax tocopherol and/or a derivative thereof.

10. The use of tocopherol and/or a derivative thereof as a stabiliser for a refined petroleum wax.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 93/03630

A. CLASSIFICATION OF SUBJECT MATTER
IPC 5 C08K5/13

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 5 C08K C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP,A,0 359 488 (NEUTROGENA) 21 March 1990 see page 2, line 41 - page 3, line 58; claims 1,7	1
A	GB,A,2 135 672 (AZUMA SHIBUE) 5 September 1984 see page 1, line 30 - line 53; claim 1	1
A	DATABASE WPI Week 7831, Derwent Publications Ltd., London, GB; AN 55811A 'ETHYLENE BASED POLYMER COMPOSITIONS' & JP,A,53 072 057 (NIPPON PETROCHEM) 27 June 1978 see abstract	1

-/--

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

'A' document defining the general state of the art which is not considered to be of particular relevance

'E' earlier document but published on or after the international filing date

'L' document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

'O' document referring to an oral disclosure, use, exhibition or other means

'P' document published prior to the international filing date but later than the priority date claimed

'T' later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

'X' document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

'Y' document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

'&' document member of the same patent family

Date of the actual completion of the international search
17 March 1994

Date of mailing of the international search report
28.04.94

Name and mailing address of the ISA
European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer
Bourgonje, A

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 93/03630

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US,A,2 619 465 (SCANLEY) 25 November 1952 see the whole document	1
A	EP,A,0 459 354 (NIPPON OIL) 4 December 1991 see claims 1,2	1
A	DE,A,11 14 319 (BASF) 28 September 1961 cited in the application	

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/EP 93/03630

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP-A-0359488	21-03-90	US-A-	4996239	26-02-91
		AU-B-	621400	12-03-92
		AU-A-	4132989	22-03-90
		JP-A-	2121915	09-05-90
		US-A-	5093107	03-03-92
		US-A-	4996238	26-02-91
GB-A-2135672	05-09-84	JP-C-	1356414	13-01-87
		JP-A-	59161372	12-09-84
		JP-B-	61024393	10-06-86
US-A-2619465		NONE		
EP-A-0459354	04-12-91	JP-A-	4031497	03-02-92
DE-A-1114319		NONE		